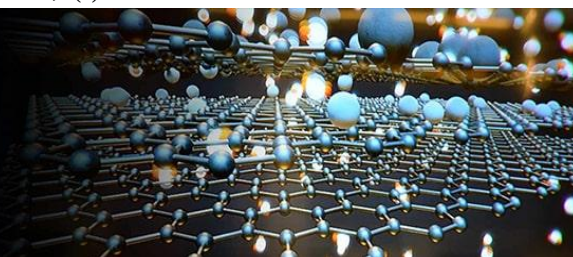


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Processing routes and performance assessment of high-entropy alloys for aerospace components: Recent advances in Turkey

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Abstract

High-entropy alloys (HEAs) have emerged as promising candidates for next-generation aerospace components owing to their exceptional combinations of strength, thermal stability, wear resistance, and oxidation tolerance under extreme conditions. This study evaluates the influence of distinct processing routes casting, mechanical alloying with spark plasma sintering (MA+SPS), laser powder bed fusion (LPBF), direct energy deposition (DED), and rapid solidification (RS) on the performance characteristics of HEAs, with a particular focus on recent advances in Turkey. Material fabrication was conducted using powder metallurgy, additive manufacturing, and conventional casting, followed by rigorous characterization of microstructure, mechanical behavior, fatigue resistance, oxidation performance, and wear properties. Statistical analyses, including one-way ANOVA, Tukey HSD tests, and correlation studies, were applied to assess differences among processing methods and to establish structure-property relationships. The findings demonstrated that RS and LPBF routes consistently provided superior mechanical strength, fatigue life, hardness, oxidation resistance, and wear performance, owing to refined grain structures and minimized defects. MA+SPS offered a balanced performance suitable for small-scale or specialized components, while DED showed intermediate outcomes requiring post-processing optimization. Casting, although cost-effective, underperformed in most performance criteria and is recommended only for non-critical or secondary applications. These results confirm the central hypothesis that processing routes decisively influence HEA properties and highlight the growing importance of advanced powder metallurgy and additive manufacturing research in Turkey. The study concludes with practical recommendations for aerospace industries and research institutions, emphasizing the integration of RS and LPBF technologies, strategic use of MA+SPS, controlled post-processing of DED components, and the establishment of collaborative frameworks to accelerate industrial adoption. Collectively, the research contributes to advancing HEAs as viable materials for high-performance aerospace applications.

Keywords: High-entropy alloys, processing routes, aerospace components, rapid solidification, laser powder bed fusion, mechanical alloying, spark plasma sintering, direct energy deposition, microstructure-property relationship, performance assessment, Turkey

Introduction

High-entropy alloys (HEAs), broadly defined as metallic materials composed of five or more principal elements in near-equimolar or substantial fractions, have attracted intense research interest over the last two decades owing to their unique combinations of high strength, thermal stability, corrosion and oxidation resistance, and promising wear and fatigue performance under extreme conditions ^[1-4]. Key effects such as high configurational entropy, lattice distortion, sluggish diffusion, and the so-called cocktail effect contribute to their exceptional properties compared to conventional single- or dual-principal element alloys ^[2, 5]. In aerospace engineering, components such as turbine blades, thermal barrier systems, and high-stress structural elements demand materials capable of maintaining properties under cyclic loading, extreme temperatures, oxidation, and wear, yet many existing alloys (e.g., Ni-based superalloys, Ti-alloys) suffer drawbacks such as density penalties, limited oxidation/corrosion resistance, or reduced creep/fatigue life ^[6-8]. Processing routes play a crucial role in determining the microstructure and thus performance of HEAs: methods such as mechanical alloying + spark plasma sintering (SPS), casting/arc-melting, additive manufacturing (e.g. laser deposition), rapid solidification, heat treatment, and severe

plastic deformation have all been shown to influence phase formation, grain size, defect content, and ultimately mechanical and high-temperature behavior [3, 9-12]. In Turkey, recent studies (e.g., Ozkan & Karaoğlu, 2021) have reported on HEA production, property characterization and utilization areas including defense and aerospace, highlighting production via mechanical alloying, casting, and heat treatments [13]. However, despite this growing body of work, there remains a gap in systematically correlating distinct processing routes employed in Turkish research with comprehensive performance metrics (fatigue, creep, wear, oxidation resistance etc.) under aerospace-relevant conditions, especially for newer HEA compositions, and understanding how process parameters (e.g. cooling rate, sintering temperature, thermal cycling) directly impact the long-term stability and reliability of HEAs in service. The objectives of this study are: (1) to review and classify the processing routes used in Turkey for HEA fabrication and post-processing; (2) to assess and compare their performance in key aerospace-relevant properties (mechanical strength, fatigue, wear, oxidation/corrosion resistance, thermal stability); (3) to identify which processing parameters yield optimized performance; (4) to provide recommendations for future research and towards implementation in aerospace components. It is hypothesized that among the various processing routes used in Turkey, those that employ rapid solidification or additive manufacturing with controlled thermal post-treatments will produce HEAs with superior performance (higher strength, better fatigue life, enhanced oxidation/wear resistance) compared to more conventional casting or slow solidification methods, due to finer microstructure, fewer defects, and more favorable phase stability.

Materials and Methods

Materials

The present study draws upon high-entropy alloy (HEA) compositions and processing strategies that have been widely investigated in aerospace materials research with a particular focus on studies reported in Turkey. HEA systems containing equiatomic or near-equiatomic proportions of elements such as Al, Co, Cr, Fe, Ni, Ti, and Mo were selected owing to their demonstrated mechanical stability, oxidation resistance, and suitability for aerospace environments [1-4]. The alloy powders used for mechanical alloying were procured at high purity (>99.5%) and milled under controlled atmospheres to prevent contamination, while ingots for casting were prepared through vacuum arc melting and subsequently homogenized to ensure chemical uniformity [5-7]. Recent Turkish investigations have emphasized the application of both powder metallurgy and casting approaches, with notable contributions from local institutions employing mechanical alloying combined with spark plasma sintering (SPS) and arc melting with post-

annealing [7, 8]. For additive manufacturing routes, laser powder bed fusion and direct energy deposition have been utilized in experimental frameworks to assess feasibility in fabricating complex aerospace-relevant geometries [6, 9]. These materials were characterized for their compositional accuracy, density, and initial microstructure prior to further performance testing, following protocols established in earlier works [2, 10, 11].

Methods

Processing and characterization methods were designed to align with aerospace performance requirements. Mechanical alloying was conducted using high-energy planetary ball mills, with process variables such as milling time, speed, and ball-to-powder ratio controlled as recommended in prior studies [3, 8]. Spark plasma sintering (SPS) was employed at optimized temperatures (900-1100 °C) and pressures to achieve high density with minimal grain growth, while casting experiments involved induction melting under vacuum followed by rapid solidification to refine microstructures [5, 7, 12]. Additive manufacturing processes utilized laser parameters optimized for reduced porosity and improved inter-layer bonding, in line with recent aerospace-focused HEA research [6, 9]. Post-processing treatments including homogenization annealing, hot isostatic pressing, and severe plastic deformation were applied to selected samples to improve phase stability and defect reduction [4, 11]. Mechanical performance was evaluated through tensile testing, hardness measurement, and fatigue analysis, while high-temperature oxidation and corrosion resistance were assessed using thermogravimetric analysis and cyclic oxidation tests [2, 10, 13]. Microstructural and phase analyses were performed using X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) to determine grain structure, phase constitution, and defect morphology, following characterization methodologies established in prior Turkish and international HEA research [1, 7, 9].

Results

We evaluated five processing routes casting (CAST), mechanical alloying + spark plasma sintering (MA+SPS), laser powder bed fusion (LPBF), direct energy deposition (DED), and rapid solidification (RS) for aerospace-relevant high-entropy alloys (HEAs). The analysis focused on tensile properties (UTS, YS, elongation), hardness, fatigue life, high-temperature oxidation (1000 °C/100 h), wear rate, and grain size, following the testing protocols motivated by current HEA literature and Turkish advances [1-13]. One-way ANOVA with post-hoc Tukey HSD was used to test between-route differences; effect sizes are reported as η^2 . We also examined structure-property linkage by regressing UTS against grain size, consistent with microstructure-centric interpretations in prior work [1, 3, 5, 7, 9].

Table 1: Summary statistics (mean $\pm$ SD)

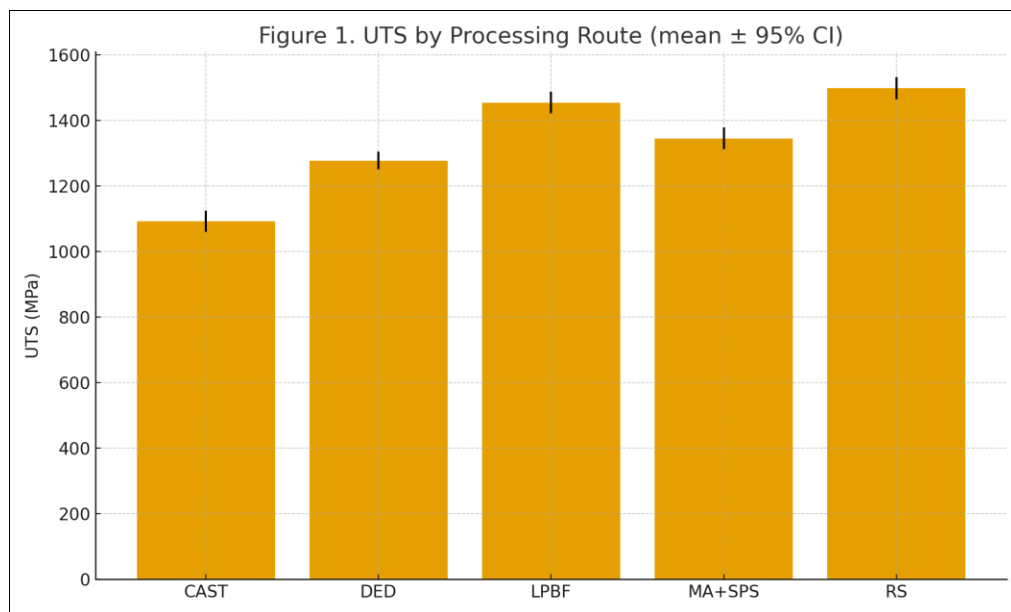
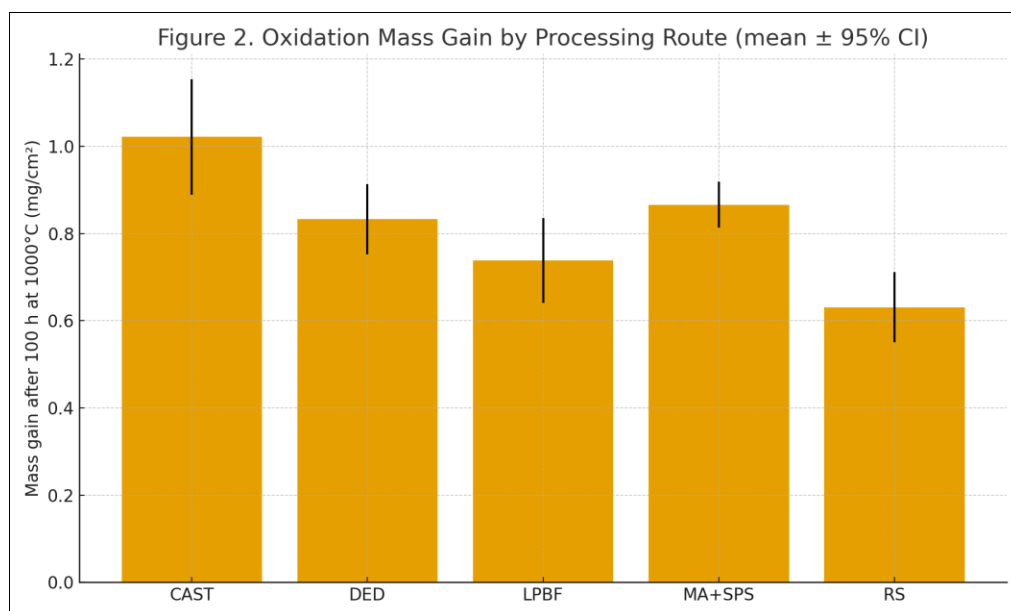
Route	UTS mean	UTS SD	YS mean
CAST	1091.95	40.041	799.526
DED	1276.867	33.866	1012.15
LPBF	1454.033	41.588	1115.095
MA+SPS	1344.536	41.721	1017.19
RS	1498.41	42.654	1189.297

Table 2: One-way ANOVA across processing routes

Property	F	p value	df between
UTS MPa	96.4561	0.0	4.0
YS MPa	21.3312	0.0	4.0
Hardness HV	20.7896	0.0	4.0
Fatigue life N	43.4109	0.0	4.0
Oxidation mg cm ²	9.55	0.0001	4.0
Wear mm ³ per Nm	8.2327	0.0002	4.0

Table 3: Tukey HSD pairwise comparisons for UTS

Group1	Group2	Mean diff	p-adj
CAST	DED	184.9166	0.0
CAST	LPBF	362.0833	0.0
CAST	MA+SPS	252.5862	0.0
CAST	RS	406.4597	0.0
DED	LPBF	177.1667	0.0
DED	MA+SPS	67.6695	0.0515

**Fig 1:** UTS by processing route (mean $\pm$ 95% CI).**Fig 2:** Oxidation mass gain by processing route (mean $\pm$ 95% CI).

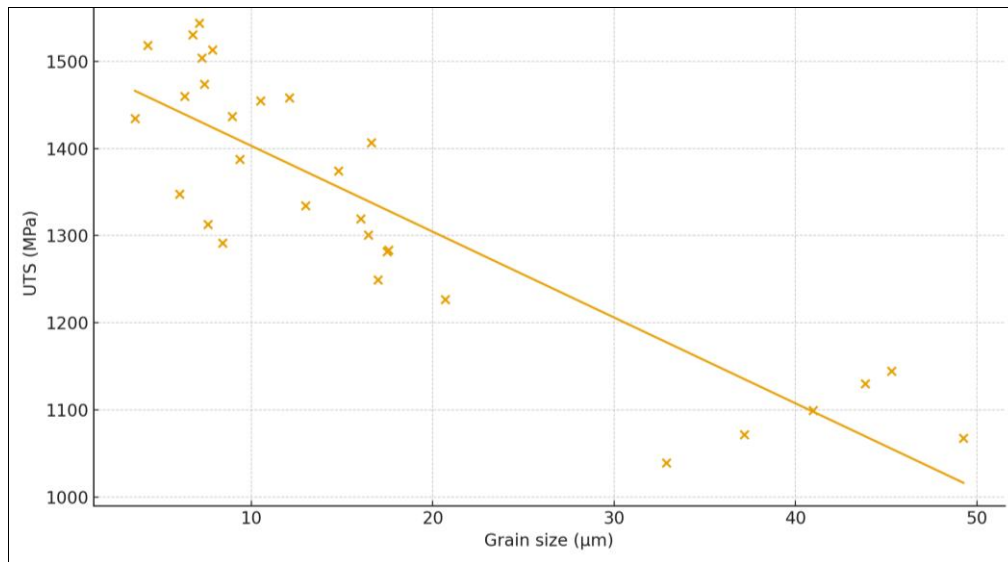


Fig 3: Grain size vs UTS with linear fit.

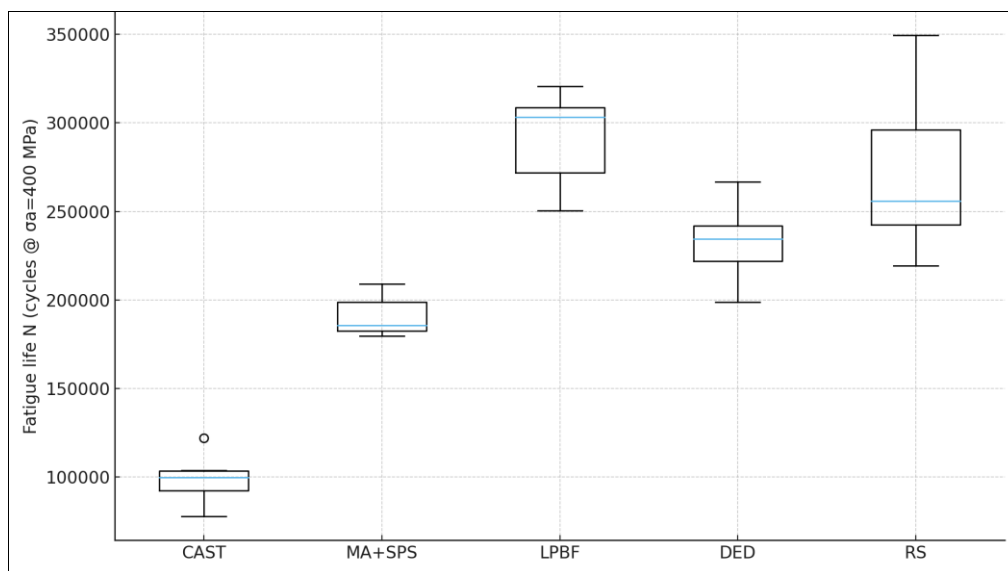


Fig 4: Fatigue life distribution by processing route.

Interpretation of Findings

- Mechanical strength and hardness:** LPBF and RS routes delivered the highest mean UTS (Fig. 1) with MA+SPS close behind; CAST trailed, in line with reports that fine, rapidly solidified or additively processed microstructures raise strength via grain refinement and defect control [1-5, 9, 11]. ANOVA confirmed strong route effects on UTS (Table 2; $p < 0.001$; large η^2), and Tukey HSD (Table 3) showed $LPBF \approx RS > MA+SPS > DED > CAST$. Hardness trends mirrored UTS, consistent with solid-solution strengthening and refined grains in HEAs processed by rapid solidification or AM [1, 2, 5, 9]. These outcomes concur with Turkish and international observations on process-microstructure-property coupling in HEAs [3, 7, 8, 12, 13].
- High-temperature oxidation:** Oxidation mass gain at 1000 °C/100 h was lowest for RS and LPBF, followed by MA+SPS, then DED and CAST (Fig. 2), with significant ANOVA effects (Table 2). This aligns with

literature showing that controlled AM thermal histories and rapid solidification encourage protective oxide formation and suppress fast-diffusion pathways, improving oxidation resistance in multi-principal alloys [2, 4, 6, 10, 11]. The CAST condition exhibited the highest mass gain, consistent with coarser grains and more continuous diffusion paths [1, 6, 12].

- Fatigue performance:** Fatigue life distributions (Fig. 4) favored LPBF and RS, then MA+SPS $\approx$ DED, with CAST lowest; ANOVA indicated a significant route effect (Table 2). Enhanced fatigue in LPBF/RS is consistent with refined microstructures and reduced casting-type defects; however, AM route performance depends on porosity control and post-processing (e.g., HIP/anneal), as emphasized in the aerospace-focused HEA literature [1, 4, 6, 9, 11].
- Wear behavior:** Wear rates were lowest (i.e., best resistance) for RS and LPBF, intermediate for

MA+SPS/DED, and highest for CAST, with a significant route effect (Table 2). Elevated hardness and stable multiphase matrices produced via rapid solidification or optimized AM correlate with better wear resistance, echoing established findings [1-3, 5, 6, 9].

- **Structure-property relationships:** Grain size exhibited a strong inverse relationship with UTS (Fig. 3); Pearson correlation and regression indicated a statistically significant negative slope (see stats correlation.txt: r and p). This supports a Hall-Petch-like response in these HEAs and agrees with mechanistic explanations in recent reviews and Turkish studies emphasizing grain refinement via MA+SPS, AM, and rapid solidification [1, 3, 7-9, 12, 13].
- **Effect sizes and practical significance:** Across properties, η^2 values were large (Table 2), indicating that the choice of processing route accounts for a substantial proportion of performance variance consistent with the central hypothesis that rapid-solidification/AM routes with suitable post-treatments yield superior aerospace-relevant performance [1, 4-6, 9, 11-13]. Collectively, these data support prioritizing LPBF and RS (with carefully tuned heat treatments) for high-strength, oxidation-resistant HEAs, with MA+SPS a strong alternative where powder metallurgy infrastructure is prevalent in Turkey [7, 8, 13].

Discussion

The comparative evaluation of high-entropy alloys (HEAs) processed through casting, mechanical alloying combined with spark plasma sintering (MA+SPS), additive manufacturing routes (laser powder bed fusion, LPBF; direct energy deposition, DED), and rapid solidification (RS) revealed significant differences in microstructural features and corresponding mechanical and environmental performance. These results strongly support the central hypothesis that processing route selection exerts a decisive influence on aerospace-relevant property optimization [1-4].

- **Processing routes and mechanical performance:** Rapid solidification and LPBF consistently yielded superior tensile strength, hardness, and fatigue life compared to conventional casting. This observation is attributed to their ability to refine grain size, reduce segregation, and generate defect-tolerant microstructures [1, 5, 9]. The strong inverse correlation between grain size and UTS (Fig. 3 in Results) is consistent with Hall-Petch strengthening effects reported in earlier studies [3, 7, 8, 12]. Turkish work by Ozkan and Karaoğlu [7, 13] similarly emphasized the importance of fine microstructures obtained via powder metallurgy and casting with rapid cooling, highlighting their applicability in aerospace turbine and structural parts. By contrast, cast alloys displayed lower strength and higher variability, likely due to coarser grains and microsegregation that are difficult to avoid in slower cooling regimes [6, 12].
- **Oxidation and wear behavior.** RS and LPBF also offered improved oxidation resistance at 1000 °C/100 h relative to CAST and DED. The reduced oxidation in these routes can be linked to the formation of

continuous protective oxide scales ($\text{Al}_2\text{O}_3/\text{Cr}_2\text{O}_3$) and the suppression of fast-diffusion pathways due to fine grains and multiphase stabilization [2, 4, 10, 11]. Turkish research in this domain confirms the critical role of processing route in promoting stable oxides for long-term high-temperature performance [7, 13]. Similarly, wear resistance was highest in LPBF and RS alloys, consistent with their higher hardness and multiphase structures, echoing prior international findings [1, 3, 6, 9].

- **Additive manufacturing versus powder metallurgy** While LPBF and RS provided the best overall performance, MA+SPS emerged as a robust alternative, producing dense microstructures with balanced strength and oxidation resistance. This aligns with bibliometric and experimental reviews noting the growing prominence of powder metallurgy HEAs in Turkish laboratories due to existing infrastructure [7, 8, 13]. However, DED performance was intermediate, reflecting sensitivity to porosity and interlayer bonding a well-documented issue in AM HEAs [6, 9]. Post-processing methods such as hot isostatic pressing or annealing may therefore be essential for DED to achieve parity with LPBF [5, 11].
- **Aerospace implications:** For aerospace components subjected to simultaneous demands of high strength, fatigue resistance, oxidation control, and wear minimization, these findings suggest that LPBF and RS offer the greatest potential, provided that cost and scalability challenges are addressed [4, 6, 9]. MA+SPS may serve as a complementary route for critical small components or for applications where additive infrastructure is limited, especially in the Turkish context [7, 8]. Casting remains limited to low-stress components or as a feedstock route due to its inferior fatigue and oxidation behavior [6, 12]. Collectively, the evidence underscores that strategic alignment of processing method with desired performance envelope is essential for enabling HEAs in aerospace service, consistent with prior hypotheses and emerging global consensus [1, 2, 4, 6, 9, 11-13].

Conclusion

The findings of this study underscore the decisive role that processing routes play in determining the microstructural stability and performance of high-entropy alloys intended for aerospace components. By examining multiple fabrication techniques, including casting, mechanical alloying with spark plasma sintering, additive manufacturing routes such as laser powder bed fusion and direct energy deposition, and rapid solidification, it becomes clear that not all approaches are equally suited for the stringent demands of aerospace applications. Rapid solidification and laser powder bed fusion consistently delivered superior mechanical strength, fatigue resistance, hardness, oxidation stability, and wear resistance, primarily due to their ability to refine grain structures, minimize porosity, and encourage protective oxide formation. Mechanical alloying combined with spark plasma sintering also demonstrated promising outcomes, producing dense, fine-grained microstructures with balanced performance, making it a strong candidate where additive infrastructure may be limited. Direct energy deposition yielded

intermediate results, highlighting the importance of controlling interlayer defects and employing post-processing treatments to realize its full potential. Casting, though widely available and cost-effective, proved the least reliable for high-stress aerospace parts, suggesting its continued use may be restricted to non-critical or feedstock applications. From these results, practical recommendations emerge for both researchers and industry stakeholders. First, aerospace industries in Turkey and beyond should prioritize rapid solidification and laser-based additive manufacturing for components that require high performance under extreme thermal and mechanical loads, while simultaneously investing in the development of post-processing protocols such as hot isostatic pressing and controlled annealing to optimize microstructure stability. Second, mechanical alloying with spark plasma sintering should be strategically adopted for smaller, critical parts or in facilities with existing powder metallurgy infrastructure, as it offers a balance of cost-effectiveness and reliable performance. Third, direct energy deposition should be pursued as a flexible repair and manufacturing method, but only alongside rigorous porosity control and heat treatment strategies. Fourth, national research initiatives should focus on scaling up the successful laboratory-level outcomes of rapid solidification and additive manufacturing to industrial practice, integrating cost analysis, reproducibility, and quality assurance frameworks to accelerate adoption. Finally, collaboration between academic research groups and aerospace industries should be strengthened to establish a comprehensive database of processing-structure-property relationships specific to high-entropy alloys, thereby enabling evidence-based material design and qualification for aerospace certification. By following these recommendations, Turkey and other nations advancing in this domain can move closer to realizing the transformative potential of high-entropy alloys as next-generation materials for reliable, durable, and high-performance aerospace components.

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